

## Amendments to the Specification

Please rewrite Paragraph sections indicated in their entirety as follows (the changes on these pages from the previous version to the rewritten version are shown with ~~striketrough~~ for deleted material and underlining for added matter):

### Page 1, Paragraph 2

Among the protein fibers, the regenerated collagen fiber exhibits a high mechanical strength like silk; and, thus, has ~~so far~~ been used in various fields. Particularly, the regenerated collagen fiber is a protein fiber maintaining a characteristic molecular structure derived from collagen and, thus, is close in drape, luster and feel to the human hair that is a natural protein fiber having complex fine structure. Such being the situation case, ~~it has been attempted there have been attempts~~ to use the regenerated collagen fiber as a replacement for human hair or in an animal hair-like fiber such as a fur (for example, see Japanese Patent Laid-Open No. 168628/1998 and Japanese Patent Laid-Open No. 168629/1998).

### Page 1, Paragraph 3 and Page 2, Paragraph 1

In general, the skin or bone of an animal is used as a raw material for the regenerated collagen fiber. The regenerated collagen fiber can be produced by treating these raw materials with an alkali or an enzyme to obtain a water-soluble collagen, followed by extruding and spinning the water-soluble collagen in an aqueous solution of an inorganic salt. Since the regenerated collagen fiber thus obtained is soluble in water, some treatments are applied thereto in order to impart resistance to water to the collagen fiber. As a method for making the regenerated collagen fiber insoluble in water, there ~~have been~~ are known methods ~~of including~~ treating the water-soluble collagen fiber with an aldehyde compound such as formaldehyde or glutaric aldehyde; ~~methods of~~ treating the water-soluble collagen fiber with metal salts such as various chromium salts, aluminum salts or zirconium salts; ~~methods of~~ treating the water-soluble collagen fiber with an epoxy compound; and ~~methods of~~ treating the regenerated collagen fiber with a combination of the above-described methods (for example, Japanese Patent Laid-Open No. 173161/1994).

Page 2 , Paragraph 2

However, being produced from collagen, the fiber produced by these methods has a lower heat resistance than that of human hair or animal hair containing keratin as a major component, and is susceptible to thermal damages (contraction in length, curling or hardening of hair tips) upon styling with a hair iron or dryer, thus ~~not being satisfactory rendering such styling unsatisfactory~~ in view of ~~hair dressing its inherent~~ properties (the term "styling" as used herein means to impart a desired form to human hair by thermal treatment in a beauty parlor or at home).

Page 3, Paragraph 1

An object of the invention is to provide a regenerated collagen fiber with excellent heat resistance, which is less apt to be damaged even ~~in styling when styled~~ with a hair iron or dryer.

Page 3, Paragraphs 2 and 3 (Combined)

#### **Disclosure Summary of the Invention**

Under such circumstances, as a result of intensive investigations, the inventors have found that the regenerated collagen fiber with excellent heat resistance can be obtained by compounding 1 to 100 parts by weight of a thermoplastic resin with 100 parts of collagen. ~~That is, Specifically, -~~the invention is embodied in a regenerated collagen fiber comprising 100 parts by weight of collagen and 1 to 100 parts by weight of a thermoplastic resin, ~~and with the thermoplastic resin is preferably being~~ obtained by polymerizing at least one member selected from the group consisting of alkyl acrylate monomers, alkyl methacrylate monomers, acrylic acid, methacrylic acid, vinyl cyanide monomers, aromatic vinyl monomers, and halogenated vinyl monomers. The invention is also embodied in a method of producing such a regenerated collagen fiber.

### Best Mode for Carrying Out the Invention

As a raw material of collagen to be used in the invention, split leather is preferred. The split leather can be obtained from a fresh raw hides or a-salted hides of animals such as cows. Such split leather ~~mostly~~ primarily comprises insoluble collagen fibers, and is usually used after removing ~~therefrom~~ flesh portions attached thereto ~~to form a network~~ and a salt component used for preventing the leather from becoming putrid or deteriorated.

### Page 4, Paragraph 2

Split leather ~~under-in~~ this condition still contains impurities; for example, lipids such as glyceride, phospholipid and free fatty acids, and proteins other than collagen, such as sugar proteins and albumin. Since these impurities greatly affect (adversely) the spinning stability in forming fiber, the quality such as luster and elongation of the resultant fiber; and the odor, it is desirable to remove these impurities in advance. ~~by~~, They may be removed, for example, ~~by~~ dipping split leather in lime to hydrolyze the fat components so as to loosen the collagen fiber, followed by applying a conventional hide treatment such as an acid-alkali treatment, an enzyme treatment and a solvent treatment.

### Page 4, Paragraph 3

The thus treated insoluble collagen is subjected to a solubilizing treatment in order to cut the crosslinking peptide portion. As such a solubilizing treatment, there may be employed an alkali solubilizing method or an enzyme solubilizing method, each of which are-is commonly employed as a ~~method of the~~ solubilizing treatment method.

### Page 5, Paragraph 1

The use of an enzyme solubilizing method is advantageous in that it is possible to obtain a regenerated collagen having a uniform molecular weight. ~~and, thus, the~~ an enzyme solubilizing method can be favorably employed in the invention. As such an enzyme solubilizing method, the methods described in, ~~for example,~~ Japanese Patent Publication No.

25829/68 or Japanese Patent Publication No. 27513/68, for example, can be employed ~~in the invention~~. Incidentally, it is possible in the invention to employ in combination both the alkali solubilizing method and the enzyme solubilizing method.

Page 5, Paragraph 2

Where additional treatments such as pH adjustment, salting-out, water wash and treatment with a solvent are applied to the collagen ~~to which the~~ after a solubilizing treatment has been applied, it is possible to obtain a regenerated collagen fiber having an excellent quality. Thus, it is desirable to apply these additional treatments to the solubilized collagen.

Pages 5, Paragraph 3 and Page 6, Paragraph 1

The solubilized collagen leather pieces thus obtained ~~is~~ are dissolved in an acidic aqueous solution having the pH value adjusted to 2 to 4.5 with hydrochloric acid, acetic acid, lactic acid or the like to provide a stock solution of a predetermined concentration. ~~of, f~~ For example, an aqueous solution of about 1 to about 15% by weight, preferably about 2 to about 10% by weight, ~~thus an aqueous solution of collagen is being prepared.~~

Page 6, Paragraph 2

~~In~~ According to the invention, a thermoplastic resin is added to either a solubilized collagen leather pieces before the acid ~~such as hydrochloric acid, acetic acid or lactic acid being~~ is added thereto, or to an aqueous solution of collagen to which the acid has been added. In either case, the resin is added in an amount of 1 to 100 parts by weight per 100 parts by weight of collagen.

Page 6, Paragraph 3

The amount of the thermoplastic resin to be compounded is preferably 3 to 80 parts by weight, and more preferably 5 to 50 parts by weight. ~~In case where~~ If the amount is less than 1 part by weight, the effect of improving heat resistance tends to become insufficient whereas, in

case where there is more than 100 parts by weight, ~~there tends to the~~ result tends to be a fragile fiber which is difficult to handle, though heat resistance ~~being is~~ improved.

Page 6, Paragraph 4

~~The Mechanism how by which~~ heat resistance improved by compounding the thermoplastic resin is not clear, but it may be presumed ~~as that~~ thermoplastic resin particles existing inside the regenerated collagen fiber form some structure within the fiber which functions to inhibit deformation such as contraction of collagen molecules upon heating with a hair iron or the like.

Page 7, Paragraph 2 and Page 8, Paragraph 1

The thermoplastic resin has a glass transition temperature of 0°C to 120°C, preferably 30°C to 100°C, and more preferably 30°C to 80°C. The term "glass transition temperature" as used herein means a middle glass transition temperature of a peak measured at a temperature-raising rate of 10°C/min according to the method described in JISK7121. In the case where the glass transition temperature is less than 0°C, the thermoplastic resin particles are liable to agglomerate upon compounding, leading to formation of large masses which reduce the strength the of resultant regenerated collagen fiber containing them. On the other hand, in the case where the glass transition temperature exceeds 120°C, effects obtained by compounding the thermoplastic resin tend to be weakened.

Page 8, Paragraph 2

Furthermore, the thermoplastic resin particles have a particle size of preferably 5 µm or less, more preferably 1 µm or less, and still more preferably 0.5 µm or less. In the case where the particle size exceeds 5 µm, there tends to result a fragile fiber. ~~As For~~ the thermoplastic resin particles, powder pulverized with a mill or latex particles prepared by emulsion polymerization or suspension polymerization may be used. In particular, latex particles obtained by emulsion polymerization are uniform in particle size and ~~have~~ has a good stability in water, ~~and, therefore, is they are~~ easy to handle; thus being preferably used.

Page 8, Paragraph 3 and Page 9, Paragraph 1

In ~~the case of~~ compounding the thermoplastic resin particles with the solubilized collagen, an acid is further added after compounding the thermoplastic resin particles, followed by ~~well~~-stirring the mixture well in a kneader or the like for 2 hours or longer, preferably 5 hours or longer, to prepare an aqueous solution of collagen wherein the particles are uniformly dispersed. In addition, in ~~the case of~~ compounding the thermoplastic resin with an aqueous solution of collagen, the mixture is ~~well~~-stirred well for 1 hour or longer in a kneader or the like to uniformly disperse the thermoplastic resin particles in the aqueous solution of collagen. These procedures are conducted at a temperature of preferably 25°C or lower. In case where the temperature is higher than 25°C, the aqueous solution of collagen might be denatured, leading to difficulty in stable production of fiber. Further, in the case of using a thermoplastic resin having a glass transition temperature of lower than 25°C, it is desirable to conduct the treatment at a temperature ~~not~~ no higher than the glass transition temperature of the added resin in order to prevent agglomeration of the resin particles.

Page 9, Paragraph 2

Additionally, the thus obtained aqueous solution of collagen may, if necessary, be subjected to a defoaming procedure by stirring under reduced pressure, or to a filtering procedure, to remove large-sized foreign matter.

Page 9, Paragraph 3

Further, to the thus obtained aqueous solution of the solubilized collagen may, if necessary, be added additives such as a stabilizer and a water-soluble high-molecular compound in proper amounts. ~~for~~ The purpose of this, for example, is improving mechanical strength, resistance to water and to heat, luster and spinning properties, preventing coloration and imparting antiseptic properties.

Page 10, Paragraph 1

The aforesaid aqueous solution of the solubilized collagen is then discharged through, for example, a spinning nozzle or slit. The discharged solution is dipped in an aqueous solution of an inorganic salt so as to obtain a regenerated collagen fiber. As the aqueous solution of an inorganic salt, ~~there may be used, for example,~~ an aqueous solution of a water-soluble inorganic salt such as sodium sulfate, sodium chloride or ammonium sulfate ~~may be used~~. Usually, the inorganic salt concentration in the aqueous solution is adjusted to 10 to 40% by weight.

Page 10, Paragraph 2

PH of the aqueous solution of the inorganic salt is desirably adjusted to 2 to 13, preferably 4 to 12, by adding a metal salt such as sodium borate or sodium acetate or hydrochloric acid, acetic acid or sodium hydroxide to the aqueous solution. In case where the pH value is smaller than 2 or exceeds 13, the peptide linkage of collagen is likely to be hydrolyzed, sometimes resulting in failure to obtain a desired fiber.

Also, it is desirable for the temperature of the aqueous solution of the inorganic salt, which is not particularly limited in the present invention, to be adjusted in general, for example, to 35°C or lower. In case where the temperature of the aqueous solution is higher than 35°C, the soluble collagen is denatured or the mechanical strength of the spun fiber is lowered, with the result that it becomes difficult to manufacture fiber thread with a high stability. The lower limit of the temperature range is not particularly limited in the invention. It suffices to adjust the lower limit of the temperature appropriately in accordance with the solubility of the inorganic salt.

Page 11, Paragraph 3 and Page 12, Paragraph 1

As the crosslinking agent, there are illustrated, for example, monoaldehydes such as formaldehyde, acetaldehyde, methyl glyoxal, acrolein, and crotonaldehyde; dialdehydes such as glyoxal, malondialdehyde, succindialdehyde, glutaraldehyde, and dialdehyde starch; alkylene oxides such as ethylene oxide and propylene oxide; halogenated alkylene oxides such as epichlorohydrin; epoxy compounds including glycidyl ethers of aliphatic alcohol, glycol and polyols, and glycidyl esters of monocarboxylic acid, dicarboxylic acid, and polycarboxylic acid;

N-methylol compounds derived from urea, melanin, acrylamide acrylic acid amide and polymers thereof; water soluble polyurethanes prepared by introducing isocyanate into a polyol or a polycarboxylic acid, followed by adding sodium hydrogen sulfite; triazine derivatives such as monochlorotriazine and dichlorotriazine; sulfate ester of oxyethyl sulfone or derivatives of vinyl sulfone; trichloropyridine derivatives; dichloroquinoxaline derivatives; N-methylol derivatives; isocyanate compounds; phenol derivatives; aromatic compounds having a hydroxyl group represented by tannin; and inorganic crosslinking agents of metal salts wherein a cation of metal such as aluminum, chromium, titanium or zirconium is combined with an anion such as sulfate ion, nitrate ion, halide ion represented by chloride ion or hydroxyl ion. However, the crosslinking agents to be used in the invention are not limited only to these ~~crosslinking agents,~~ and ~~o~~Other crosslinking agents may also be used which can reduce contraction with hot water, water absorption or swelling degree in water of the regenerated collagen and can make the regenerated collagen fiber insoluble in water. Additionally, water-insoluble crosslinking agents may be used as an emulsion or a suspension. These crosslinking agents may usually be used alone or as a mixture of two or more of them.

Page 13, Paragraph 2

Drying is usually conducted in a hot air convection dryer. The regenerated collagen fiber is liable to contract upon being dried, and it is extremely difficult for the once deformed collagen fiber to be formed into a desired form. Thus, in the invention, drying is conducted in a state wherein the fiber is fixed at both ends under tension or in a stretched state wherein a load is applied to both ends of the fiber so that the contraction ratio of the fiber after drying becomes 30% or less, preferably 20% or less, and still more preferably 10% or less without being broken. In case where the contraction ratio of the fiber thread after drying exceeds 30%, complicated unevenness tends to be formed on the surface of the fiber to cause detrimental influences on touch feel. The atmospheric temperature within the dryer is not particularly limited, but a temperature of not lower than the glass transition temperature of the added thermoplastic resin is preferred because the effect of improving heat resistance is more remarkable. This may be attributed to ~~that~~ a continuous structure is being formed within the regenerated collagen fiber by welding of the added thermoplastic resin particles to each other, which serves to improve heat resistance. Further, as to the atmospheric temperature within the dryer, it is preferably 100°C or

lower, and more preferably 90°C or lower, because, in case where it is too high, the fiber might be colored or denatured. Drying time is longer than that which is required to completely dry the fiber and shorter than that at which decoloration of the fiber becomes serious.

Page 14, Paragraph 2

The water wash is intended to prevent precipitation of an oiling agent caused by a salt and to prevent the salt from being precipitated from the regenerated collagen fiber during drying within a drying machine. In the case where the salt is precipitated, the regenerated collagen fiber is cut or broken, and the formed salt scatters within the drying machine so as to be attached to the heat exchanger within the drying machine, leading to a low heat transfer coefficient. Also, the oiling is effective for preventing the fiber from hanging up in the drying step and for improving the surface state of the regenerated collagen fiber.

Page 14, Paragraph 3

The thus obtained regenerated collagen fiber containing the thermoplastic resin has an excellent heat resistance, and enables ~~to conduct~~ styling with a hair iron or dryer to be conducted with ~~maintaining the drape that a natural protein fiber has, thus being being maintained.~~ The fiber is, accordingly, more favorably usable as a substitute or a piece for improving human hair and animal hair.

Page 14, Paragraph 4

The invention is now described in more detail by reference to Examples, ~~which,~~ However, the examples do not limit the invention in any way.

Page 23, Paragraph 1

The invention is embodied in a method for improving heat resistance of the regenerated collagen fiber, which makes the regenerated collagen fiber into an extremely excellent product to be used as a substitute of human hair, for example, wig or hair piece, or head-decorating products such as doll hair. It is also embodied in a heat resistant regenerated collagen fiber.